

Supporting information

RESEARCH ARTICLE

Surface molecularly imprinted polymers for solid-phase extraction of (–)-epigallocatechin gallate from toothpaste

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Abstract Surface molecularly imprinted polymers (SMIPs) have been synthesized to selectively determine (–)-epigallocatechin gallate in aqueous media. SMIPs were prepared using a surface grafting copolymerization method on a functionalized silica gel modified with β -cyclodextrin and vinyl groups. The morphology and composition of the SMIPs were investigated by scanning electron microscopy, Fourier transform-infrared spectroscopy and thermogravimetric analysis. In addition, the molecular binding capacity, recognition properties and selectivity of the SMIPs were evaluated. The imprinted polymers were found to have a highly specific recognition and binding capacity for (–)-epigallocatechin gallate in aqueous media which is the result of the hydrophobic properties of the β -cyclodextrin and the hydrogen-bonding interactions of methacrylic acid. The SMIPs were successfully employed as solid-phase extraction adsorbents prior to the HPLC determination of (–)-epigallocatechin gallate in toothpaste. The HPLC analysis had a linear dynamic range of 0.5–50.0 $\mu\text{g} \cdot \text{mL}^{-1}$ with a correlation coefficient of 0.9998 and the recoveries ranged from 89.4% to 97.0% with relative standard deviations less than 4.8%. The limit of detection and limit of quantification were 0.17 and 0.33 $\mu\text{g} \cdot \text{mL}^{-1}$, respectively. The method provides a promising approach for the preparation of selective materials for the purification and determination of complex samples.

Keywords β -cyclodextrin, (–)-epigallocatechin gallate, surface molecular imprinting, solid-phase extraction

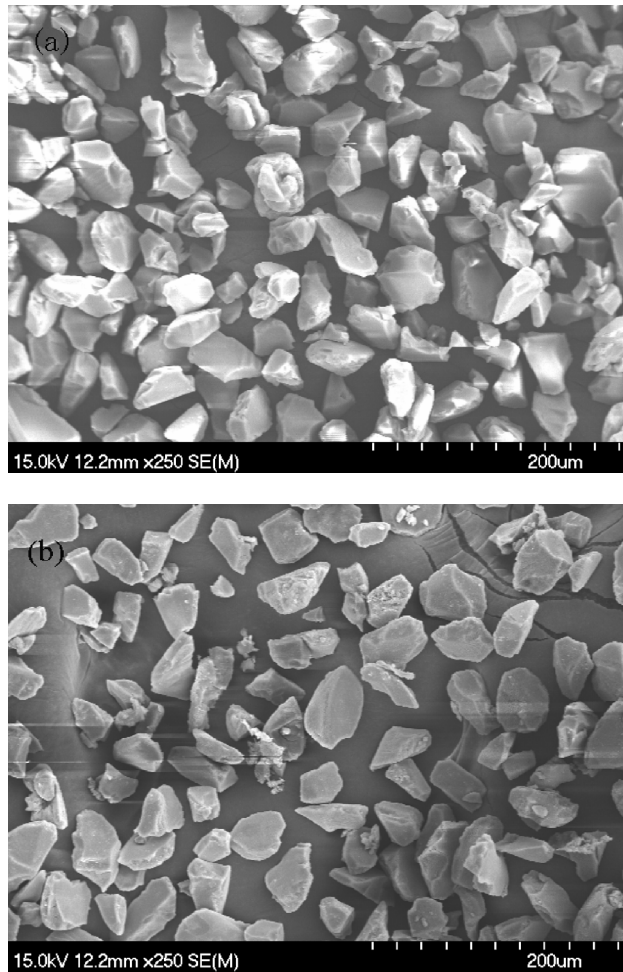


Fig. S1 SEM microphotographs of activated silica gel (a) and SMIP1 (b)

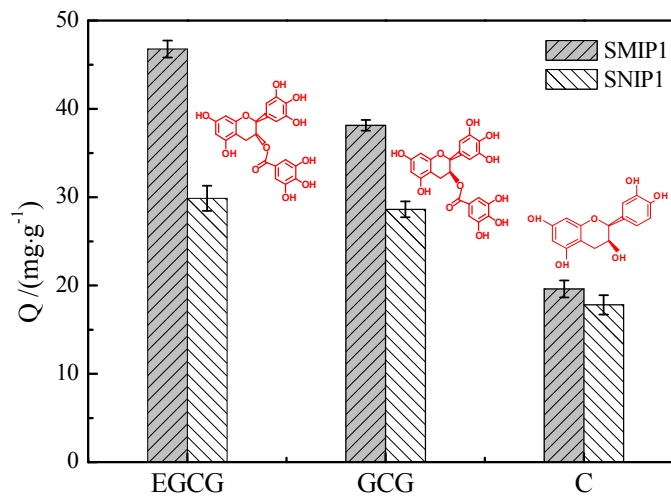


Fig. S2 The specific recognition capability of SMIP1 and SNIP1 for EGCG and two structurally similar compounds

Table S1 Comparison of some methods used for determination of EGCG

Matrix	Treatment method	Detection	LOD /(ng·mL ⁻¹)	LOQ /(ng·mL ⁻¹)	Recovery /%	Ref.
Human plasma	–	HPLC-CEAD	2.8 ^{e)}	5.0 ^{c)}	96.5	[1]
Rat plasma	–	UPLC-ESI-MS	–	10	68.5–86.5	[2]
Green tea	SE ^{a)}	HPLC-UV	30 ^{d)}	80 ^{d)}	–	[3]
Human plasma	Ionic liquid	Sweeping-MEKC	0.4 ^{d)}	1.2 ^{d)}	100–100.5	[4]
Teas	MIES ^{b)}	Cyclic-voltammetry	73.3 ^{e)}	–	98.6–102.2	[5]
Apple	SE ^{a)}	HPLC-UV	–	–	96–105	[6]
Rat plasma	-	HPLC-UV	125	500	85.73–91.93	[7]
Toothpaste	MISPE	HPLC-DAD	170	330	89.4–97.0	This work

a) SE represents solvent extraction; b) MIES represents molecularly imprinted electrochemical sensor; c) LOD is defined as the lowest concentration significantly different from zero, and LOQ is the lowest concentration significantly different from LOD; d) $LOQ = 10S_a/b$ and $LOD = 3S_a/b$, where S_a is the standard deviation of the intercept of the calibration curve and b is the slope of the calibration curve; e) $LOD = (3\delta)/s$, where δ indicates the standard deviation of the seven consecutive measurements of the detection solution in the absence of EGCG, and s is the sensitivity obtained from the slope of the calibration curve

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